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R. B. Clark[a]; S. K. Zeto[a]; K. D. Ritchey[a]; V. C. Baligar[a]

[a] Appalachian Farming Systems Research Center, Agricultural Research Service, U.S. Department of Agriculture, Beaver, WV, U.S.A.

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MINERAL ACQUISITION BY MAIZE GROWN IN ACIDIC SOIL AMENDED WITH COAL COMBUSTION PRODUCTS

R. B. Clark,* S. K. Zeto, K. D. Ritchey, and V. C. Baligar

Appalachian Farming Systems Research Center,
U.S. Department of Agriculture, Agricultural Research
Service, 1224 Airport Rd., Beaver, WV 25813-9423

ABSTRACT

Large amounts of coal combustion products (CCPs) are produced when coal is burned for generation of electricity. Some of these CCPs could potentially be used as soil amendments, and information about their effects on plant mineral nutrition is needed. Glasshouse experiments were conducted to test the effects of different levels of 15 CCPs and chemical grade $CaCO_3$, $CaSO_4$, and $CaSO_3$ added to acidic soil (Umbric Dystrochrept) on shoot calcium (Ca), sulfur (S), phosphorus (P), potassium (K), magnesium (Mg), manganese (Mn), iron (Fe), zinc (Zn), copper (Cu), and aluminum (Al) concentrations of maize (*Zea mays* L.). The CCPs consisted of two fly ashes (FAs), one CaO material, three fluidized bed combustion products (FBCs), three "non-stabilized" flue gas desulfurization products (FGDs), three "stabilized" FGDs, and three "oxidized" (FGD gypsum) FGDs. Level of CCP added to soil ranged from beneficial to detrimental effects on plants. Differences in shoot mineral element

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*Corresponding author. E-mail rclark@afsrc.ars.usda.gov

concentrations were related to kind and amount of CCP added and soil pH. Plants grown in unamended (pH 4) soil had symptoms of P and Mg deficiencies and Al toxicity. High concentrations of Ca, S, Mg, and Mn accumulated when plants were grown with some CCPs, but most mineral nutrients were at concentrations considered normal for maize. Shoot concentrations of P, K, Zn, Mn, Fe, and Al decreased when soil pH became high (>7). Even though detrimental mineral element acquisition effects were imposed on plants at high levels of CCP application, shoot element concentrations were usually normal when applied at levels near those commonly used as soil amendments.

INTRODUCTION

When coal is burned for generation of electricity, large amounts of coal combustion products (CCPs) are produced (97.8 million metric tons in 1998) (1). CCPs make up the third largest source of mineral resources in the United States after crushed stone and sand/gravel and ahead of portland cement and iron ore (S.S. Tyson, ACAA, personal communication). CCPs could be a valuable mineral resource for many uses, including as soil amendments. Use of CCPs as soil amendments, although small compared to the total amount produced, could be important in overall management of these products. Most CCPs present relatively little risk to the environment at levels commonly used as soil amendments (2, 3). Soils have extensive buffering and/or diluting effects on these materials and plants usually receive benefits when these products are added to soil at appropriate levels (4–11).

Application of CCPs to soil may raise soil pH and help mitigate acidic soil problems such as plant mineral toxicities (Al and Mn) and deficiencies (P and Mg). CCPs may also serve as sources of essential mineral nutrients to plants, particularly Ca, S, Mg, and B, and improve soil physical properties, which would indirectly improve mineral nutrient acquisition by plants (2, 3, 8, 9, 11–15). These products may also provide nutrients and enhance organic materials (composts) in landscape, mine spoil, and nursery use (2, 10, 16, 17) and reduce mineral transport, especially P, from surface soils into streams and estuaries or into leachates (18–21).

An important reason for applying CCPs to soil is to provide mineral nutrients to plants and to alleviate toxicities commonly associated with acidic soils. Certain CCPs are more effective than others in providing mineral nutrients and mitigating acidic soil problems, and some CCPS may be detrimental if too much is applied. For example, fly ashes (FAs) and fluidized bed combustion

products (FBCs) normally have high pH to provide extensive neutralizing potential [$CaCO_3$ equivalency (CCE)], but may also have high soluble salts [e.g., Ca, S, sodium (Na), and boron (B)] and high amounts of trace elements [e.g., B, arsenic (As), selenium (Se), molybdenum (Mo), cadmium (Cd), chromium (Cr), nickel (Ni), and lead (Pb)] to induce undesirable toxicities (2, 3, 9). In addition, plants grown in acidic soil amended with CCPs may develop mineral deficiencies (e.g., P, Zn, Fe, Mn) if too much is added and soil pH is too high (>7–8). Use of FAs and FBCs in soil and ability of these products to provide minerals to plants and to induce and/or alleviate mineral toxicities has received considerable attention (2, 3, 9).

In contrast to FAs and FBCs, flue gas desulfurization products (FGDs) generally do not have as high pH, contain high Ca and S, but lower soluble salts, and may or may not contain high amounts of trace elements (3, 11). Mineral element toxicities and/or deficiencies have been induced on plants when certain FGDs were applied to soil at relatively low levels (5, 6). These responses have usually occurred with FGDs that have been oxidized to become FGD gypsum products. The FGD "scrubber sludges" often contain extensive amounts of FA, $CaCO_3$, and/or CaO, which are often added prior to discarding or using the final product. Thus, reactions of many FGD may be similar to those of FAs and the materials added rather than "non-stabilized" or "oxidized" FGDs. Since FGDs are relatively new CCPs, information about mineral nutrition of plants grown in soil amended with FGDs is scarce.

Bermuda grass [*Cynodon dactylon* (L.) Pers.] and alfalfa (*Medicago sativa* L.) grown in the field acquired high S when grown in soil with relatively high rates of FGD, but not to concentrations exceeding maximum tolerance levels (~4 g kg^{-1}) for animal intake (22, 23). Perennial ryegrass (*Lolium perenne* L.) had decreased P when grown in a glasshouse with 10 and 20 metric ton ha^{-1} FGD (4). Of several CCPs added to soil for growth of annual ryegrass (*Lolium multiflorum* Lam.) in a glasshouse, FGD gypsum was the most benign of all and increased root growth without elevating concentrations of trace elements (24). Maize (*Zea mays* L.) grown in soil mixed with 8 and 10% FGD in a greenhouse had B, As, and Se concentrations above normal, while other minerals remained at concentrations normal for plants (25). Addition of FGD gypsum to a pasture increased Mn and S and decreased Mg in forage (K.D. Ritchey, unpublished data).

The objectives of our study were to determine potential use of CCPs in acidic soil for growth and mineral nutrition of plants. This phase of the study concerned mineral acquisition by maize. Maize was chosen for the study because of its fast growth pattern so that many CCPs at various levels could be evaluated in a relatively short period of time. Levels of CCPs used gave beneficial and detrimental effects on plant growth and mineral nutrition to provide information about limits for adding CCPs to acidic soil.

MATERIALS AND METHODS

An acidic Porters (coarse-loamy, mixed, mesic, Umbric Dystrochrept) soil collected from eastern Tennessee was used in each experiment. Some properties of the soil before addition of amendment and fertilizer were 81% sand, 14% silt, and 5% clay; 13.2% organic matter (Walkley-Black digestible); 3.88 pH_{Ca} (1 soil:1 10 mM $CaCl_2$), 4.22 pH_W (1 soil:1 water), and 0.09 dS m^{-1} electrical conductivity (EC) (1 soil:1 water); 2.70 mg P kg^{-1} soil (Bray-1-extractable); 0.74 mg B kg^{-1} soil (hot-water extractable); 0.18 K, 0.10 Mg, 0.24 Ca, and 0.03 Na in cmol$_c$ kg^{-1} soil (1 M ammonium acetate-extractable); 86.0 Fe, 3.86 Mn, 1.20 Zn, and 0.09 Cu in mg kg^{-1} soil (5 mM DTPA-extractable); and 5.38 cmol$_c$ Al kg^{-1} soil (81% Al saturation) and 6.11 cmol$_c$ total acidity kg^{-1} soil (1 M KCl-extractable). Methods of soil analysis for the various elements/properties are described in Page et al. (26).

The 15 CCPs used in experiments were: two FAs [FA-12 (Class F) and FA-18 (Class C)]; one CaO product (CaO-10); three FBCs (FBC-15, FBC-21, and FBC-26); three "non-stabilized" FGDs (FGD-1, FGD-4, and FGD-6); three "stabilized" FGDs (FGD-2, FGD-5, and FGD-8); and three "oxidized" FGDs (FGD-16, FGD-22, and FGD-27). "Non-stabilized" means FGDs with no materials added to stabilize/ solidify/ reduce water content, "stabilized" means added stabilizing/solidifying/drying materials (e.g., $CaCO_3$, CaO, $Ca(OH)_2$, or other drying material) to "non-stabilized" FGDs, and "oxidized" means FGDs that have been converted to $CaSO_4$ (FGD gypsum) from $CaSO_3$ through forced air or other oxidizing methods. Chemical properties and mineral element concentrations in the CCPs have been reported (5). Since CCPs often contain extensive amounts of $CaCO_3$, $CaSO_4$,[1] and $CaSO_3$, these compounds (chemical grade) were added to soil separately at different levels to evaluate their effects on plant mineral acquisition similar to those of the CCPs.

Air-dried soil was passed through a 2-mm screen before treatments were added. Each CCP or chemical grade compound and fertilizer (100 mg N kg^{-1} soil as NH_4NO_3 and 505 mg K and 400 mg P kg^{-1} soil as KH_2PO_4) were thoroughly mixed with soil. Levels of each CCP or control compound were different to meet impact expectations on growth, and are listed in the tables. Deionized water was added to soil mixes to provide near water holding capacity and soils were equilibrated 7 d before being placed in pots (1.0 kg soil mix pot^{-1}) for plant growth. Because of the large number of CCPs, control compounds, and different levels of each used, several experiments were conducted over time. The different CCPs and control compounds were included in repeat experiments. Each

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[1]$CaSO_4$ has been used throughout this article rather than the $CaSO_4$-water containing compounds, especially gypsum ($CaSO_4 \times 2H_2O$).

experiment consisted of completely randomized blocks with four replications. Unamended soil was used as the control for each product used.

Seeds of the maize hybrid PA329 × PA353P were surface sterilized with 0.1-strength NaOCl (household bleach) for 5 min, rinsed thoroughly with deionized water, and germinated between wrapped germination papers moistened with deionized water containing dilute $CaSO_4$ to assure good root development. Three 3-day-old seedlings were transplanted to each pot. Deionized water was added manually as needed to avoid splashing on stalks and leaves, to provide sufficient moisture for plant growth, and to avoid leaching from pots. Minor leaching occurred from some pots where plants did not grow well, and soil pH and EC values after plant growth were similar to those at the beginning of the experiment.

Experiments were conducted in a glasshouse ($25 \pm 3°C$) using natural as well as artificial light to extend short days to 14-h light and provide extra light during cloudy days. High-pressure sodium lamps provided artificial light at $400-500 \, \mu mol^{-2} \, s^{-1}$ at plant height.

Plants were harvested after they had grown in treated soil for 21 d (24-d-old plants). This was because plants grown in unamended soil and with high levels of some CCPs were near death, and this short growth period was used so data from poor growing plants could be included in the data. Shoots were severed $\sim$1 cm above the soil surface, dried, weighed, and ground to pass a 0.5-mm screen in preparation for mineral element analysis. Soil/roots from pots was placed on a 2-mm screen, representative soil samples collected for pH and EC determinations, and roots thoroughly washed free of soil, blotted dry, and crowns cut from roots. Both roots and crowns were dried separately, and weighed. Soil pH (1 soil:1 10 mM $CaCl_2$) and EC (1 soil:1 water) were determined using pH and EC electrodes.

Samples of dried ground shoot material were weighed ($50-100$ mg) into Teflon containers, digestion solution (1.7 mL 15.8 M HNO_3 + 0.2 mL 11.4 M HCl + 0.1 mL 28.9 M HF) was added, and containers were placed in microwave digestion bombs (Parr Instrument Co., Moline, IL[2]). These samples were microwave digested 4 min at 70% power followed by 2 min at full power (635 W), allowed to cool in the microwave 5 min, and removed to cool at ambient temperature. Digested solutions were brought to a final volume of 10.0 mL with distilled deionized water. Solutions were filtered and analyzed for mineral elements by inductively coupled plasma spectroscopy (Model 3580, Applied Research Laboratories, Dearborn, MI[2]). If digested

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[2]Mention of trade names or commercial products in this publication is solely for the purpose of providing specific information and does not imply recommendation or endorsement by the U.S. Department of Agriculture.

Table 1. Whole Plant DM, Soil pH, and Shoot Mineral Concentrations of Maize Grown in Acidic Soil Amended with Different Levels of Control Compounds $CaCO_3$, $CaSO_4$, and $CaSO_3$

Material	Level	DM	pH	Ca	S	P	K	Mg	Mn	Fe	Zn	Cu	Al
	% in soil	mg plant^{-1}				g kg^{-1}					mg kg^{-1}		
$CaCO_3$ (lime)	0	427	3.94	1.30	1.96	1.30	24.8	0.80	266	88	26.5	1.7	53
	0.09	587	4.07	3.54	2.29	1.29	22.9	0.63	168	71	22.1	2.7	23
	0.18	694	4.17	4.94	2.08	1.30	32.5	0.46	152	68	32.0	2.5	21
	0.27	636	4.19	4.86	2.37	1.34	29.0	0.64	128	83	23.6	3.4	23
	0.45	662	4.56	6.34	2.60	1.44	32.5	0.59	144	92	29.7	3.3	20
	0.91	827	5.22	8.57	2.49	1.30	32.5	0.70	129	100	20.4	3.4	19
	1.82	682	6.03	10.30	2.51	1.25	25.2	0.79	133	101	8.2	4.2	8
LSD (P = 0.05)		58	0.08	0.43	0.14	0.08	1.5	0.03	7	8	2.2	0.6	4
$CaSO_4$ (low)	0	466	3.94	1.52	1.89	1.16	27.2	0.81	296	52	33.4	0.7	24
	0.25	285	4.00	5.70	6.82	1.66	17.7	0.82	238	60	21.5	0.3	57
	0.5	189	4.05	6.60	6.70	2.28	14.4	1.08	219	62	20.0	3.8	77
	1	216	4.06	5.92	6.98	2.10	17.5	1.15	227	76	24.8	2.6	76
	2	313	4.08	7.20	10.32	1.62	26.4	1.17	262	72	31.4	2.6	53
	4	374	4.15	6.60	8.37	1.40	30.3	1.46	256	86	36.5	3.4	36

LSD (P = 0.05)		45	0.04	0.46	0.57	0.08	0.8	0.06	17	11	2.0	0.9	5
CaSO$_4$ (high)	0	368	4.00	1.22	2.43	1.92	34.5	0.91	236	80	27.6	2.0	42
	5	392	4.29	7.30	8.98	2.11	43.4	1.62	560	89	44.8	2.8	34
	10	516	4.36	5.99	6.88	2.11	45.5	2.14	452	91	44.5	3.0	31
	25	579	4.78	4.86	5.61	2.31	47.8	3.14	295	90	33.9	3.7	16
	50	409	5.77	4.23	5.89	2.54	51.5	5.20	224	95	20.9	4.3	9
	75	360	6.54	7.34	9.18	2.53	50.4	3.92	115	71	13.1	3.8	11
LSD (P = 0.05)		53	0.03	0.44	0.70	0.13	1.7	0.18	25	6	3.0	0.4	9
CaSO$_3$	0	466	3.94	1.52	1.89	1.16	27.2	0.81	296	52	33.4	0.7	24
	0.25	307	4.10	7.45	7.46	1.86	20.3	0.90	759	82	24.6	2.1	63
	0.5	231	4.12	7.03	6.91	2.30	22.4	0.91	704	70	25.4	1.6	63
	1	222	4.17	6.96	4.97	2.78	30.2	0.83	711	67	35.7	1.8	47
	2	136	4.29	6.39	3.88	3.36	26.3	0.89	463	50	36.1	0.8	32
	4	61	4.49	5.77	4.66	5.68	14.8	1.93	407	34	29.2	1.5	28
LSD (P = 0.05)		44	0.02	0.42	0.44	0.21	2.0	0.21	43	15	4.1	1.2	5

samples had to be stored before analysis, they were placed in plastic containers and stored at $-10°C$.

Data were analyzed statistically using analysis of variance procedures, and least significant differences (LSD) at $P = 0.05$ were used to determine differences among means for each product used.

RESULTS AND DISCUSSION

Data on shoot and root dry matter (DM) and soil pH and EC have been published (5). Since mineral availability in soil and acquisition by plants are so highly dependent on soil pH, whole plant DM and soil pH data have been listed in the tables as reference. Except for one CCP (FA-18 with 6.5 dS m^{-1} EC), soil EC at highest treatment levels of CCPs did not exceed values (3.5 dS m^{-1}) that would be detrimental to moderately salt sensitive plants (27). Shoot B and trace element concentrations for plants grown with these CCPs have also been published (28, 29), and these data have not been included in tables. Calcium and S concentrations were presented in a proceedings article (30) that is relatively unaccessible to scientists because of the proprietary nature of the publisher, so these data have been included in the tables.

Plants grown in unamended soil and sometimes in soil mixed with control substances and many of the CCPs developed P, Mg, and Zn deficiencies, with P and Mg deficiencies occurring more frequently than Zn deficiency. Additional P was required for plants to grow well in this acidic soil because of low P (2.7 mg kg^{-1} soil), which was below that considered needed to support growth of many plants ($>$12 mg P kg^{-1} soil) (31). Added P was also needed to overcome P deficiency in other studies where this soil had been used (7, 32). Symptoms of Al toxicity were also observed on some plants grown in unamended soil.

Information on mineral element concentrations of plants grown in amended soil with the control compounds CaCO$_3$, CaSO$_4$, and CaSO$_3$ are presented first to better define and understand CCP effects on mineral acquisition, since many of the CCPs used contained high amounts of these compounds. The low (deficiency) and high (excessive or toxic) concentrations of the various mineral elements presented in the text are those reported for young maize shoot tissue (33, 34).

CaCO$_3$, CaSO$_4$, and CaSO$_3$ Effects

Shoot P concentrations in plants grown in unamended or CaCO$_3$-amended soil ranged from 1.25–1.44 g kg^{-1} (Table 1), which were below P concentrations

considered adequate for maize ($>2.0\,\text{g}\,\text{kg}^{-1}$). Phosphorus deficiency was common for plants grown with $CaCO_3$-amended soil. Plants grown in soil amended with $>0.25\%$[3] levels of $CaSO_4$ or any level of $CaSO_3$ had P concentrations considered adequate for maize. This may have been because of reduced growth, so that plants did not need as much P as the faster growing plants grown with $CaCO_3$.

Shoot Mg concentrations for maize grown in unamended soil were below those considered adequate ($>1.0\,\text{g}\,\text{kg}^{-1}$), and Mg deficiency was commonly observed for plants grown in not only unamended soil but in $CaCO_3$-, $CaSO_4$-, and $CaSO_3$-amended soil (Table 1). Magnesium deficiency was also common in studies where maize was grown in this soil without amendment (7), and is common for many plants grown in acidic soil (35). Shoot Ca was at concentrations in these treatments that could interact with Mg and induce Mg deficiency. Once soil Ca:Mg molar ratios were about 30–50:1, Mg deficiency on maize was alleviated (7).

Zinc deficiency developed on some plants grown in this amended soil, but not as frequently as Mg and P deficiencies. On occasion when soil pH >7, Fe deficiency was observed. Concentrations <10–$15\,\text{mg}\,\text{Zn}\,\text{kg}^{-1}$ and $<50\,\text{mg Fe}$ kg^{-1} in leaves are considered to be below adequacy for maize.

Some plants grown in this unamended soil exhibited symptoms of Al toxicity, even though shoot Al concentrations of plants grown in unamended soil (Table 1) did not approach those considered toxic for maize ($>200\,\text{mg}\,\text{kg}^{-1}$). Another potential mineral element toxicity associated with amending this acidic soil was that of Mn. Concentrations of Mn in shoots approached or exceeded those considered to be toxic ($>500\,\text{mg}\,\text{kg}^{-1}$) when plants were grown in soil amended with $CaSO_3$ and high $CaSO_4$ (Table 1).

Maize grown in $CaSO_4$- and $CaSO_3$-amended soil generally had increased shoot Ca and S concentrations as amount of material added to soil increased, although S tended to decrease at the highest levels of added $CaSO_3$ (Table 1). However, Ca did not reach excessive limits ($>10\,\text{g}\,\text{kg}^{-1}$) when plants were grown with $CaSO_4$ and $CaSO_3$, while S approached excessive limits ($>5\,\text{g}\,\text{kg}^{-1}$) with added $CaSO_4$ and $CaSO_3$. Both Ca and S were above deficiency concentrations (1.0 Ca and 0.8 S in $\text{g}\,\text{kg}^{-1}$) for plants grown in unamended soil. For maize grown with $CaCO_3$-, $CaSO_4$-, and $CaSO_3$-amended soil, shoot K, Mn, Fe, and Cu concentrations were generally within limits considered adequate (>20–26 K in $\text{g}\,\text{kg}^{-1}$ and >15 Mn, >50 Fe, and >2–5 Cu in $\text{mg}\,\text{kg}^{-1}$) for maize.

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[3]To convert percentage values of added amendment in this paper to metric ton ha^{-1} multiply by 22 and to convert to US ton acre^{-1} multiply by 10.

Table 2. Whole Plant DM, Soil pH, and Shoot Mineral Element Concentrations of Maize Grown in Acidic Soil Amended with Different Levels of FA and CaO

CCP[†]	Level	DM	pH	Ca	S	P	K	Mg	Mn	Fe	Zn	Cu	Al
	% in soil	mg plant^{-1}				g kg^{-1}					mg kg^{-1}		
FA-12 (Class F)	0	449	3.91	1.15	1.54	0.91	17.3	0.67	208	76	20.5	0.7	44
	1	695	4.02	3.65	2.20	1.04	27.2	1.12	232	79	32.3	1.3	22
	2	741	4.04	4.83	2.02	1.12	28.6	1.43	214	71	35.8	0.3	17
	3	861	4.13	5.43	3.09	1.16	28.0	1.68	185	73	34.9	0.6	20
	5	676	4.44	7.10	3.49	1.36	31.2	2.36	170	78	38.9	0.6	24
	10	536	4.82	8.72	4.60	1.94	32.1	3.45	119	65	26.2	0.2	20
LSD (P = 0.05)		69	0.05	0.35	0.17	0.14	2.0	0.14	12	5	4.6	0.5	3
FA-18 (Class C)	0	480	4.03	1.95	1.98	0.74	16.4	0.68	310	128	19.3	2.9	70
	0.5	985	4.64	6.80	1.90	0.56	27.8	1.89	145	100	32.8	5.4	47
	1	952	5.06	8.24	2.56	1.24	27.9	4.04	132	112	23.0	7.0	40

2	1002	5.43	9.50	2.76	1.20	26.2	3.91	134	105	26.6	8.4	37
3	1009	5.88	10.46	3.39	1.43	24.6	6.28	122	117	25.6	8.2	32
5	904	6.33	10.94	3.70	1.68	24.6	7.80	114	104	22.4	10.6	33
10	1111	7.52	10.87	3.11	1.41	22.7	7.75	68	83	14.4	8.1	25
25	993	7.60	12.80	3.48	1.11	14.1	9.19	12	90	10.4	6.2	31
LSD (P = 0.05)	115	0.05	0.44	0.15	0.05	1.2	0.25	7	12	2.2	0.6	7
CaO-10												
0	449	3.91	1.15	1.54	0.91	17.3	0.67	208	76	20.5	0.7	44
1	794	6.34	9.46	2.22	1.06	25.2	2.46	69	94	9.7	3.4	16
2	638	7.71	13.22	2.97	1.03	23.7	3.28	41	155	8.4	4.2	21
3	493	8.14	14.44	3.00	1.12	22.3	3.86	23	158	8.1	2.5	20
5	418	8.41	12.78	1.85	1.30	12.1	3.99	10	134	6.9	3.4	18
10	55	9.82	8.17	1.83	6.16	11.9	3.00	14	55	33.5	5.4	48
LSD (P = 0.05)	86	0.28	0.53	0.16	0.37	1.6	0.19	8	17	2.7	1.1	9

[†] FA-12 = BP-#12, FA-18 = BP-#18, and CaO-10 = BP-#10 as designated in (5).

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Fas, CaO, and FBCs

Both Ca and S concentrations increased in shoots as level of FA-12 added to soil increased, and these elements did not approach excess concentrations (Table 2). FA-12 (Class F FA) contained less Ca and S than FA-18 (Class C FA) and several other CCPs. Shoot concentrations of P and Mg increased as level of FA-12 increased in soil (Table 2), but shoot P concentrations remained below those considered adequate for maize and shoot Mg at the lowest level of FA added was near the deficiency limit. Phosphorus deficiency symptoms were observed on plants grown at most levels of FA-12. Low shoot P and P deficiency symptoms were not expected since FA-12 contained relatively high P. Apparently the P in this FA was not in an available form, and may have been present as Ca-P precipitates. Potassium, Fe, Zn, Cu, and Al concentrations remained relatively constant in shoots of plants grown with different levels of FA-12 added to soil, while Mn decreased about 2-fold with high levels of FA-12 (Table 2). Even so, each of these mineral elements were considered adequate for maize.

Shoot Ca concentrations became high and approached excessive limits, when plants were grown with high levels of FA-18, while shoot S concentrations remained relatively normal (Table 2). Like plants grown with FA-12, shoot P was relatively low and near or below deficiency limits. Slight P deficiency symptoms appeared on plants grown in soil with each level of added FA-18, even though DM was relatively high. High soil pH values such as those noted when FA-18 was added could potentially induce deficiencies of P, Mn, Fe, and Zn (3). Magnesium concentrations increased extensively and became relatively high with added FA-18, and approached excessive limits ($>8.0\,g\,kg^{-1}$). FA-18 was Class C FA which contained high Mg, and enhanced foliar Mg concentration was expected. When soil pH was >7 (two highest levels of FA-18), shoot K, Mn, Fe, Zn, Cu, and Al concentrations decreased and this could have lead to mineral deficiencies. At low levels of application where soil pH was <7, shoot concentrations of these elements were adequate for maize.

Plant responses often differ when FAs are added to soil, and some plants have enhanced mineral element acquisition while other plants have opposite responses (2, 3). If added FAs contain high amounts of Ca, S, and Mg, concentrations of these mineral nutrients should be higher in plants grown in amended compared to unamended soil. A major concern for FA addition to soil is plant acquisition of excess or toxic concentrations of B and trace elements (2, 3). Boron accumulated to excess when plants were grown with high levels of FA (28), but trace elements were below standard limits and normal for plant tissue (29).

Calcium oxide [$CaO/Ca(OH)_2$] is a material commonly added to many CCPs, especially wet "scrubber sludge" FGDs, to solidify and stabilize these

Table 3. Whole Plant DM, Soil pH, and Shoot Mineral Element Concentrations of Maize Grown in Acidic Soil Amended with Different Levels of FBCs

FBC[†]	Level	DM	pH	Ca	S	P	K	Mg	Mn	Fe	Zn	Cu	Al
	% in soil	mg plant^{-1}				g kg^{-1}					mg kg^{-1}		
FBC-15	0	606	4.00	1.26	1.80	0.99	25.4	0.58	238	66	26.4	0.6	31
	0.5	1156	4.88	7.14	2.20	0.98	30.5	0.72	120	72	28.3	1.7	17
	1	1122	5.70	9.41	2.33	1.03	30.6	1.22	109	93	22.8	1.2	26
	2	822	7.09	12.53	2.93	1.07	31.6	1.89	90	241	8.8	3.5	34
	3	602	7.61	14.01	3.34	1.09	33.0	1.67	63	192	7.9	2.5	36
	5	459	8.19	18.72	3.85	1.01	17.9	1.12	27	121	7.9	0.8	69
LSD (P = 0.05)		99	0.05	0.82	0.17	0.07	1.3	0.07	10	13	2.2	0.4	10
FBC-21	0	567	4.03	1.90	1.98	0.74	16.4	0.68	310	128	25.6	2.9	70
	0.5	1168	4.72	5.50	4.53	0.68	25.8	5.21	162	105	38.9	3.6	47
	1	1263	5.26	6.04	4.30	1.14	24.6	7.68	117	104	27.6	3.3	31
	2	1023	6.08	6.80	4.53	1.42	22.3	9.47	125	127	27.6	4.2	32
	3	1199	6.54	6.42	4.38	1.28	20.2	12.71	112	100	18.6	4.2	26
	5	815	6.74	6.15	4.02	1.42	17.3	18.01	102	113	14.7	3.9	27
	10	635	7.19	4.27	3.91	1.91	7.0	21.54	33	95	12.1	2.1	25
	20	343	8.25	4.44	3.92	3.88	4.8	21.33	9	76	12.8	3.3	28
LSD (P = 0.05)		68	0.05	0.26	0.23	0.10	1.1	0.42	6	13	2.0	0.3	3
FBC-26	0	251	3.82	1.08	2.10	2.29	21.0	1.16	741	85	66.2	1.8	90
	0.5	319	4.60	9.09	4.60	3.16	35.7	0.84	320	113	58.8	1.4	35
	1	71	5.12	10.20[‡]	5.41[‡]	7.21[‡]	31.5[‡]	5.35[‡]	335[‡]	633[‡]	842[‡]	558[‡]	216[‡]
	2.5	32	6.78	ND[§]	ND	ND	ND	ND	ND	ND	ND	ND	ND
	5	28	7.80	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	10	33	8.68	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	20	49	10.40	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
LSD (P = 0.05)		14	0.27	1.04[¶]	0.54[¶]	0.30[¶]	1.4[¶]	0.49[¶]	20[¶]	22[¶]	27.1[¶]	4.52[¶]	48.2[¶]

[†] FBC-15 = BP-#15, FBC-21 = BP-#21, and FBC-26 = BP-#26 as designated in (5).
[‡] Only one sample had sufficient tissue for analysis.
[§] ND = not determined because of insufficient tissue for analysis.
[¶] Comparison of 0 and 0.5% levels of FBC-26 only.

products. Soil pH increased to highest of values (~10) when 10% CaO was added to soil, which was likely due to high CCE (130%). Shoot Ca concentrations in plants grown with >2% CaO-10 approached excessive limits, while shoot S concentrations remained adequate to low (Table 2). Except at the highest level of CaO-10 added where plants were unhealthy and shoot mineral concentrations were altered extensively, shoot P concentrations were below critical limits for maize and P deficiency symptoms were prevalent. When soil pH was >7, shoot K, Mn, Fe, and Zn concentrations decreased compared to plants grown in lower pH soil. Except at the highest level of CaO-10, most mineral elements were adequate for maize grown with this material.

Added FBC-15 increased shoot Ca concentrations to excess, while plants grown with FBC-21 had shoot Ca concentrations considered adequate (Table 3). Shoot S concentrations were not excessive and were adequate for plants grown with varied levels of both of these FBCs. Shoot P concentrations were below adequacy for plants grown with both of these FBCs, and most of the other mineral elements, except Mg, were adequate for maize. Plants grown with varied levels of FBC-21 in soil had consistently higher Mg concentrations, even reaching excessive concentrations ($>8.0 \, \text{g kg}^{-1}$), when added at levels greater than 2%. Shoot K, Mn, and Zn were low and decreased to near deficiency concentrations when high levels of FBC-15 and FBC-21 were applied to soil (Table 3). This likely occurred because of high soil pH.

Severe detrimental effects on growth occurred with relatively low levels (0.5%) of FBC-26 (Table 3). Except at the highest levels (10 and 20%), increases in soil pH were not sufficiently high to impose the detrimental effects noted on plants grown with this CCP. Nevertheless, plants grown with FBC-26 added to soil at >0.5% were so small that tissue was insufficient for mineral analysis. In the one sample where sufficient DM was obtained for analysis (plants grown with 1% FBC-26), shoot concentrations of the elements were so high that detrimental effects would be expected.

The FBCs normally contain high Ca [usually as anydrite ($CaSO_4$), unreacted sorbent ($CaCO_3$), and $CaO/Ca(OH)_2$] and S, and usually have high pH (often ~12) (36). Combinations of high Ca, particularly $CaO/Ca(OH)_2$ and $CaSO_4$ may be useful as neutralizers for soil acidity and sources of soluble Ca salts for plants. However, pozzolanic activity [ability to solidify (cement)] could create problems if FBCs are used at high levels (9). This was an advantage when FBC was applied within rows as a cap in established apple (*Malus domestica* Borkh.) orchards to provide mineral nutrients (Ca and Mg), reduce weed growth, and preserve water (36). Apple leaves had higher Mg the first couple of years after FBC had been applied, but decreased over time due to high Ca or as Ca transported throughout the soil. Increases in plant Ca, Mg, and S were noted for alfalfa (*Medicago sativa* L.) grown with FBC amended soil in a glasshouse (37), and enhanced Mg, S, and Mo were noted in maize and alfalfa grown with Mg

Table 4. Whole Plant DM, Soil pH, and Shoot Mineral Element Concentrations of Maize Grown in Acidic Soil Amended with Different Levels of "Non-stabilized" FGDs

FGD[†]	Level	DM	pH	Ca	S	P	K	Mg	Mn	Fe	Zn	Cu	Al
	% in soil	mg plant^{-1}				g kg^{-1}					mg kg^{-1}		
FGD-1	0	466	3.94	1.52	1.89	1.16	27.2	0.81	296	52	33.4	0.7	24
	0.5	524	4.13	6.34	5.88	1.30	33.2	1.34	536	59	41.3	0.9	18
	1	552	4.19	6.06	4.35	1.45	35.9	1.82	495	62	42.2	1.2	12
	2	366	4.38	6.88	3.60	2.26	33.2	3.40	509	93	44.7	1.2	16
	3	187	4.53	4.02	4.04	3.38	27.5	4.53	430	93	30.2	0.4	12
	5	124	4.94	5.77	3.92	4.86	16.8	4.97	273	34	24.5	<0.1	11
LSD (P = 0.05)		41	0.09	0.34	0.30	0.21	1.6	0.17	20	9	3.6	0.6	2
FGD-4	0	449	3.91	1.15	1.54	0.91	17.3	0.67	208	76	20.5	0.7	44
	0.5	472	4.06	7.38	4.78	1.44	26.4	2.03	593	75	49.4	2.0	32
	1	213	4.34	7.03	3.86	2.57	20.8	2.90	387	80	26.9	4.2	38
	2	156	4.59	7.72	4.41	3.57	16.7	3.36	327	61	22.2	3.3	58
	3	126	5.06	8.37	4.95	4.26	12.5	4.59	274	59	21.7	3.1	57
	5	47	5.62	3.93	4.86	8.35	10.2	3.96	106	52	45.8	5.8	69
LSD (P = 0.05)		51	0.28	0.57	0.40	0.28	0.9	0.36	33	6	3.2	0.7	12
FGD-6	0	606	4.00	1.26	1.80	0.99	25.4	0.58	238	66	26.4	0.6	31
	1	807	4.10	6.96	6.23	1.16	28.7	1.37	682	95	52.3	2.5	42
	2	692	4.19	6.68	3.95	1.17	31.2	1.84	628	88	54.5	5.3	36
	3	472	4.29	6.99	3.77	1.74	37.3	2.66	533	104	53.0	5.0	39
	5	275	4.42	7.71	4.80	2.77	31.1	4.04	520	103	46.4	2.0	47
	10	238	4.64	7.75	4.74	3.29	26.2	4.85	366	84	34.8	1.5	37
LSD (P = 0.05)		61	0.05	0.30	0.49	0.10	1.7	0.17	30	9	5.1	0.9	6

[†] FGD-1 = BP-#1, FGD-4 = BP-#4, FGD-6 = BP-#6 as designated in (5).

Table 5. Whole Plant DM, Soil pH, and Shoot Mineral Element Concentrations of Maize Grown in Acidic Soil Amended with Different Levels of "Stabilized" FGDs

FGD[†]	Level	DM	pH	Ca	S	P	K	Mg	Mn	Fe	Zn	Cu	Al
	% in soil	mg plant^{-1}				g kg^{-1}					mg kg^{-1}		
FGD-2	0	466	3.94	1.52	1.89	1.16	27.2	0.81	296	52	33.4	0.7	24
	0.5	662	4.24	5.79	3.30	1.24	34.0	2.03	420	55	42.3	0.5	13
	1	580	4.44	6.04	2.50	1.41	35.9	2.73	322	58	41.7	1.2	4
	2	172	5.13	7.15	3.57	3.32	24.5	4.88	327	100	23.4	0.7	9
	3	86	5.52	6.26	3.53	4.61	19.9	5.78	155	54	30.6	1.9	12
	5	95	6.46	8.76	4.25	5.27	15.3	7.51	87	25	26.1	1.2	9
LSD (P = 0.05)		47	0.09	0.34	0.26	0.38	1.4	0.27	14	8	4.3	0.6	3
FGD-5	0	449	3.91	1.15	1.54	0.91	17.3	0.67	208	76	20.5	0.7	44
	0.5	543	3.97	6.72	4.50	1.16	26.1	1.26	485	80	42.0	2.0	29
	1	631	4.17	7.45	4.17	1.35	26.8	2.00	467	78	42.8	1.9	30
	2	515	4.38	8.63	3.17	1.81	28.1	2.51	396	81	29.1	2.2	31
	3	365	4.97	9.20	2.96	2.31	27.8	3.37	287	78	20.0	1.8	24
	5	69	5.93	9.08	5.46	6.65	14.5	4.43	133	46	29.9	4.1	47
LSD (P = 0.05)		67	0.28	0.64	0.25	0.39	2.1	0.24	17	6	4.6	0.6	6
FGD-8	0	606	4.00	1.26	1.80	0.99	25.4	0.58	238	66	26.4	0.6	31
	1	942	4.23	6.27	4.75	1.04	31.2	1.10	425	78	45.9	3.0	24
	2	948	4.29	7.84	3.98	1.17	34.6	1.32	413	79	46.0	3.6	21
	3	1130	4.58	7.35	2.43	1.17	34.8	2.04	317	69	38.8	3.5	22
	5	825	4.78	8.14	2.57	1.41	39.8	2.66	314	105	39.8	2.6	36
	10	511	5.38	10.39	2.85	1.80	38.2	3.71	242	90	27.7	2.8	34
LSD (P = 0.05)		111	0.05	0.71	0.17	0.07	1.8	0.19	22	8	3.7	1.0	5

[†] FGD- = BP-#2, FGD-5 = BP-#5, FGD-8 = BP-#8 as designated in (5).

Table 6. Whole Plant DM, Soil pH, and Shoot Mineral Element Concentrations of Maize Grown in Acidic Soil Amended with Different Levels of "Oxidized" (Gypsum Quality) FGDs

FGD[†]	Level	DM	pH	Ca	S	P	K	Mg	Mn	Fe	Zn	Cu	Al
	% in soil	mg plant^{-1}				g kg^{-1}					mg kg^{-1}		
FGD-16	0	333	4.00	1.59	2.13	0.99	25.4	1.04	278	108	28.7	2.6	45
	5	337	4.07	7.86	8.62	1.38	25.5	1.23	322	74	29.5	2.8	74
	10	339	4.18	8.15	8.16	1.30	24.4	1.71	422	86	37.4	3.2	60
	25	469	4.26	6.95	5.06	1.20	26.0	2.56	508	96	38.4	3.9	43
	50	636	4.65	5.13	2.56	1.16	28.0	3.16	489	93	27.4	4.7	36
	75	582	5.52	4.84	2.29	1.26	27.0	4.43	341	78	20.5	4.2	25
LSD (P = 0.05)		79	0.06	1.30	0.53	0.09	1.4	0.17	26	15	3.6	0.4	9
FGD-22	0	333	4.00	1.53	2.23	2.10	23.9	1.23	298	100	29.8	2.6	65
	5	439	4.29	8.35	5.02	2.12	34.7	0.64	376	94	47.3	4.8	31
	10	521	4.36	8.58	3.63	1.88	34.9	1.16	234	101	39.2	5.0	26
	25	574	4.78	10.75	2.64	1.84	30.9	2.11	131	122	23.3	4.8	19
	50	453	5.77	11.95	2.48	2.08	31.0	2.92	107	182	10.6	5.3	13
	75	460	6.54	9.74	2.14	2.23	31.8	2.31	67	102	9.8	5.4	13
LSD (P = 0.05)		50	0.06	0.54	0.18	0.12	1.3	0.11	13	13	3.6	0.7	7
FGD-27	0	251	3.82	1.08	2.10	2.29	21.0	1.16	741	85	29.8	1.8	90
	1	677	4.22	5.19	2.52	2.15	34.2	2.74	253	102	45.9	1.4	38
	2.5	653	4.60	4.01	2.55	2.28	35.7	4.40	166	101	44.5	1.4	36
	5	528	5.38	3.90	2.63	2.72	34.7	6.90	133	100	36.4	1.5	30
	10	446	6.40	4.37	2.94	2.87	31.6	11.36	126	105	29.4	1.3	31
	25	263	7.50	4.93	3.76	2.77	26.0	16.20	104	104	49.7	2.6	25
	50	114	8.30	3.73	3.97	2.58	15.9	15.86	103	71	81.6	2.2	34
LSD (P = 0.05)		30	0.27	0.31	0.36	0.15	0.8	1.05	15	11	5.3	0.2	17

[†] FGD-16 = BP-#16, FGD-22 = BP-#22, FGD-27 = BP-#27 as designated in (5).

enriched FBC in the field (38). FBC-26 which had such detrimental effects on maize at levels $>0.5\%$ raised soil pH dramatically, had the highest soluble salts and Ca concentrations of all CCPs tested, and had low K and Mg.

FGDs

High SO_3-S was likely a major factor affecting mineral acquisition and for decreasing DM of maize grown with "non-stabilized" FGDs (FGD-1, FGD-4, and FGD-6). However, SO_3-S can be readily oxidized to SO_4-S (days or weeks) under appropriate conditions (39, 40), and it is not known how much SO_3-S may have oxidized to SO_4-S during the incubation and growth periods of our experiments. Since these products enhanced growth at low levels, it was assumed that much SO_3-S had been oxidized to SO_4-S. These FGDs also increased soil pH moderately as level in soil increased (Table 4). Except for plants grown in unamended soil and/or with the lowest levels of these FGDs, shoot Ca, S, P, K, Mg, Fe, Zn, and Cu concentrations were generally adequate and Al concentration was not excessive for maize. At low to moderate levels of these FGDs added to soil, shoot Mn concentrations increased to where they might be considered excessive ($>500\,mg\,kg^{-1}$). Increased Mn availability may have occurred in soil when the SO_3-S in these FGDs was being oxidized to SO_4-S.

The "stabilized" FGDs (FGD-2, FGD-5, and FGD-8) used (Table 5) were the same initial products as those in Table 4, only stabilizing agents had been added. Shoot concentrations of the mineral elements followed patterns similar to those noted for the "non-stabilized" FGDs, except that Ca, P, and Mg were higher and S was generally lower for plants grown with "stabilized" compared to "non-stabilized" FGDs (Tables 4 and 5). Mineral nutrient acquisition also followed patterns similar to that of plant growth. That is, plant DM was enhanced when these "stabilized" high $CaSO_3$ FGDs were added to soil at low levels, with FGD-8 having more positive effects over broader levels compared to FGD-2 and FGD-5 (Table 5). "Stabilized" FGDs generally had greater positive effects on mineral acquisition than did their counter "non-stabilized" FGDs (Tables 4 and 5). These results indicated that Ca, P, and Mg were introduced into the "stabilized" FGDs from materials that had been added. Fly ash and CaO are commonly added to FGDs as stabilizing materials, and these products often contain relatively high amounts of mineral elements. From data of the FAs used in our study, high amounts of mineral nutrients could have been added to FGDs if they had been added as stabilizing materials.

Concentrations of many mineral elements in plants grown with varied levels of the "oxidized" FGD-16 had patterns similar to plants grown with chemical grade $CaSO_4$ (Tables 1 and 6). That is, FGD-16 did not increase, and

sometimes decreased, growth when added at $>10\%$ levels to soil, which indicated that FGD-16 was likely a high grade $CaSO_4$ compound. Low levels of added FGD-16 did not improve growth likely because of Al toxicity from Ca replacement of Al on soil exchange sites and/or Ca induction of Mg deficiency. Similar responses were noted in other studies (6, 7). FGD-16 was generated specially as a wallboard quality FGD gypsum, and did not appear to contain many mineral elements commonly added to other FGDs. In contrast, FGD-22 (also a FGD gypsum) enhanced mineral acquisition and plant DM at the lowest levels used (Table 6), indicating that FGD-22 had received some added mineral elements sometime during its production. Plants grown with FGD-16 had shoot P concentrations below adequate, and plants grown with FGD-22 had P concentrations just above or near adequacy. Enhanced P deficiency has also been reported when continued or high levels of $CaSO_4$ have been added to soil (41). The other mineral nutrients in plants grown with these "oxidized" FGDs were at relatively normal concentrations.

Plants grown in this acidic soil amended with the lowest level of FGD-27 (1%) had not only improved mineral nutrition, but higher DM ($\sim$3-fold) over plants grown in unamended soil (Table 6). These plants had mineral nutrient concentrations closer to adequacy, especially Ca, S, and P, and greatest growth enhancements of any plants grown with CCPs used in our studies. FGD-27 was a FGD+Mg product [$\sim$6% $Mg(OH)_2$] and it enhanced plant DM at low soil levels, indicating that Mg was a major factor inhibiting plant growth in this unamended acidic soil. Reduced acquisition of Ca in plants grown with FGD-27 may have been partially due to the high Mg which could interact with and decrease Ca. Shoot Mg increased extensively as level of FGD-27 increased in soil, and reached concentrations near excessive ($>8.0\,\mathrm{g\,kg^{-1}}$) at the highest levels applied. Boron accumulation was also excessive when plants were grown with high levels of FGD-27 (28), and was likely a major factor reducing plant growth at the highest levels added. The other mineral elements in shoots were adequate for maize, except at the highest level of FGD-27 where soil pH was high and concentrations of some minerals decreased (K, Mn, and Fe) (Table 6).

Silicon can also be fairly high in CCPs, but Si did not accumulate in shoots to any extent (highest shoot Si concentration for any level of added CCP was $77\,\mathrm{mg\,kg^{-1}}$), changes were small, and Si often was below detectable limits. Accordingly, shoot Si data have not been reported.

CONCLUSIONS

Maize grown in acidic soil amended with CCPs received benefits when applied at appropriate levels. Plants received benefits when levels of FA, FBC,

and CaO were low ($\sim 1-2\%$), while plants receiving benefits from FGDs were at higher levels. This was primarily because FGDs are high $CaSO_4$ products, and plants can usually tolerate high $CaSO_4$ before detrimental effects occur. Level of FGD which benefitted plants depended on stabilizing materials or other salts added and sometimes on SO_3-S still remaining in the products. Added Mg into FGDs was especially beneficial since plant growth was restricted in this acidic soil by Mg deficiency. High Ca also induced Mg deficiency. Phosphorus in CCPs was not readily available for plant use. Many CCPs applied at relatively low levels improved soil pH sufficiently to alleviate Al toxicity, while high levels added increased soil pH to such high values that many mineral nutrient decreased and deficiencies would likely be induced. Plants grown with "stabilized" FGDs had mineral acquisition patterns similar to those of FA, FBC, and/or CaO (only diluted), which indicated that these materials were introduced into the final products. Plants grown with "oxidized" FGDs responded similar to those receiving gypsum or relatively pure $CaSO_4$. Shoot Ca and S concentrations also became high in plants grown with high levels of some FGDs. When CCPs were added at high levels, high Ca concentrations in plants were associated with low Mg, and S concentrations were near or above maximum tolerance limits for animal intake (42).

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